In Vitro Shear Bond Strength of Three Selfadhesive Resin Cements and a Resin-Modified Glass Ionomer Cement to Various Prosthodontic Substrates

Camila Sabatini • Manthan Patel • Eric D'Silva

Clinical Relevance

Self-adhesive resin cements demonstrated superior bond strength to a variety of prosthodontic substrates relative to resin-modified glass ionomer cement, indicating that they are able to provide a wider array of clinical applications. However, selection of the cement should be determined largely by the type of substrate and setting reaction.

SUMMARY

Objective: To evaluate the shear bond strength (SBS) of three self-adhesive resin cements and a resin-modified glass ionomer cement (RMGIC) to different prosthodontic substrates.

Materials and Methods: The substrates base metal, noble metal, zirconia, ceramic, and

*Camila Sabatini, University at Buffalo, Restorative Dentistry, Buffalo, NY, USA

Manthan Patel, University at Buffalo, Buffalo, NY, USA Eric D'Silva, University at Buffalo, Buffalo, NY, USA

*Corresponding author: 3435 Main Street, 215 Squire Hall, 215 Squire Hall, Buffalo, NY 14214; e-mail: cs252@buffalo.

DOI: 10.2341/11-317-L

resin composite were used for bonding with different cements (n=12). Specimens were placed in a bonding jig, which was filled with one of four cements (RelyX Unicem, Multilink Automix, Maxcem Elite, and FujiCEM Automix). Both light-polymerizing (LP) and selfpolymerizing (SP) setting reactions were tested. Shear bond strength was measured at 15 minutes and 24 hours in a testing device at a test speed of 1 mm/min and expressed in MPa. A Student t-test and a one-way analysis of variance (ANOVA) were used to evaluate differences between setting reactions, between testing times, and among cements irrespective of other factors. Generalized linear regression model and Tukey tests were used for multifactorial analysis.

Results: Significantly higher mean SBS were demonstrated for LP mode relative to SP mode (p<0.001) and for 24 hours relative to 15 minutes (p < 0.001). Multifactorial analysis revealed that all factors (cement, substrate, and setting reaction) and all their interactions had a significant effect on the bond strength (p < 0.001). Resin showed significantly higher SBS than other substrates when bonded to RelyX Unicem and Multilink Automix in LP mode (p<0.05). Overall, FujiCEM demonstrated significantly lower SBS than the three selfadhesive resin cements (p < 0.05).

Conclusions: Overall, higher bond strengths were demonstrated for LP relative to SP mode, 24 hours relative to 15 minutes and self-adhesive resin cements compared to the RMGICs. Bond strengths also varied depending on the substrate, indicating that selection of luting cement should be partially dictated by the substrate and the setting reaction.

INTRODUCTION

The long-term success of indirect restorations depends on several factors, including an adequate design, preparation, and selection of the restorative material. An aspect equally important to the longevity of indirect restorations is the integrity of the bonded interface between the tooth and the restoration. Currently, most resin cements use an etch-andrinse or a self-etch adhesive in combination with a low-viscosity dual polymerizing resin cement. However, this multi-step bonding procedure is complex, technique sensitive, and it involves significant chair time. A new generation of self-adhesive resin cements has been developed recently that eliminates the need for etching, priming, and bonding as separate steps. These self-adhesive resin cements are based on new monomer, filler, and initiator formulations. The acidic monomer replaces the previous three steps by combining the use of adhesive and cement into a single application. These multi-functional phosphate-based acidic methacrylates can react with the basic fillers in the luting cement and the hydroxyapatite of the hard tooth tissue.² Self-adhesive resin cements combine the high-strength and low-solubility advantages of resin cements with the characteristic ease of use of selfadhesive systems, making them highly attractive to the clinician.

Evidence is limited as to how the bond strength of newer self-adhesive resin cements compares to that of conventional self-adhesive resin-modified glass ionomer cements (RMGICs) when bonded to a variety of prosthodontic materials under multiple testing conditions. While RMGICs are self-adhesive and provide simultaneous fluoride release, aspects such as water absorption with the associated setting expansion, potential for crack development, and less associated esthetics make these cements less than ideal for situations such as the cementation of all-ceramic crowns.3 Recent studies have shown higher bond strengths for self-adhesive resin cements compared to RMGICs when bonded to a variety of materials such as noble and non-noble alloys, zirconia, aluminum oxide ceramic, and pressable ceramic.^{4,5}

Different studies have reported on the bond strength of self-adhesive resin cements to enamel and dentin, 1,2,6-9 as well as different substrates such as alloys, ¹⁰⁻¹³ ceramics, ^{4,14} and polymers. ¹⁵ However, most of these studies concentrate on a single substrate, type of setting reaction, or testing time. As self-adhesive cements continue to gain popularity for the cementation of indirect restorations, large comparative studies are needed to gain a better understanding of the overall behavior of these cements under multiple testing conditions and when bonded to a variety of prosthodontic substrates.

Therefore, the objective of this study was to evaluate the shear bond strength (SBS) of three dual polymerized self-adhesive resin cements and a RMGIC to a variety of prosthodontic substrates (base metal, noble metal, zirconia, ceramic, and resin composite). Furthermore, this study aimed to evaluate differences in SBS values between 15 minutes and 24 hours and between self-polymerizing (SP) and light-polymerizing (LP) setting reactions for the different cement-substrate combinations. The null hypothesis was that there would be no significant difference in mean SBS among the tested cements, between SP and LP modes, and between 15 minutes and 24 hours.

MATERIALS AND METHODS

Bonding Substrates

The bonding substrates, including commercial names and composition are summarized in Table 1. One hundred ninety-two specimens were prepared for each of the following substrates: base metal, noble alloy, densely sintered yttrium-stabilized zirconia, lithium disilicate glass ceramic, and resin composite. For the metallic substrates, noble metal rectangular pieces (15 mm long × 5 mm wide × 1 mm high) and base metal cylindrical blocks (10 mm diameter and 5 mm thick) were used. The original

Self-adhesive Cements							
Group	Туре	Composition	Lot No.	Manufacturer 3M ESPE (St Paul, MN, USA)			
RelyX Unicem	Dual polymerized self-adhesive resin cement	55%-65% Glass powder 15%-25% Methacrylated phosphoric acid esters 10%- 20% TEGDMA 1%-5% Silane-treated silica 1%-5% Sodium persulfate	337810				
Multilink Automix	Dual polymerized adhesive resin cement with self- etching primer	22%-26% Dimethacrylates 6%-7% HEMA <1% Benzoyl peroxide 40% Barium glass, YF ₃ , spheroid mixed oxide	L27890	Ivoclar Vivadent (Schaan Liechtenstein)			
Maxcem Elite	Dual polymerized self-adhesive resin cement	19%-40% Methacrylate esther monomers Other—inert mineral fillers, activators stabilizers, colorants, YF ₃	3100070	Kerr Corporation (Orange, CA, USA)			
FujiCEM Automix	Resin-modified glass ionomer cement	30%-40% Polyacrylic acid 30%-40% Distilled water 2% Silica powder 20% Silicone dioxide 2%-3% Benzensulfonic acid sodium salt	0404091	GC America (Alsip, IL, USA)			
Bonding Substrate	es						
Base metal	Identalloy	Co 60% Cr 30% Other 10%		Ivoclar Vivadent (Schaan Liechstenstein)			
Noble metal	Harmony Medium	Au 77% Ag 13% Cu 8% Other 2%		Ivoclar Vivadent (Schaan Liechstenstein)			
Zirconia	IPS e.max ZirCAD	87% ZrO ₂ , Y ₂ O ₃ , HfO ₂ , Al ₂ O ₃		Ivoclar Vivadent (Schaan Liechstenstein)			
Ceramic	IPS e.max CAD	$>$ 57% $\rm{SiO}_2,\rm{Li}_2\rm{O},\rm{K}_2\rm{O},\rm{P}_2\rm{O}_5,\rm{ZrO}_2,\rm{ZnO},$ $\rm{Al2O}_3,\rm{MgO}$ and pigments		Ivoclar Vivadent (Schaan Liechstenstein)			
Composite resin	Z100	80%-90% Silane-treated ceramic 1–10% BisGMA 1–10% TEGDMA <1% 2- Benzotriazolyl-4-methylphenol		3M ESPE (St Paul, MN, USA)			

zirconia and ceramic blocks were cut using a low-speed saw (Isomet, Buehler, Lake Bluff, IL, USA) to obtain square blocks ($10~\text{mm} \times 10~\text{mm} \times 2~\text{mm}$). The zirconia specimens were sintered following manufacturer's recommendations. The composite specimens were fabricated using a ring-shaped mold (10~mm diameter and 2~mm height) and light polymerized.

All substrates were embedded in a chemically polymerized methacrylate (Fastray, HJ Bosworth, Skokie, IL, USA). The exposed surfaces were

sequentially polished with 320-, 400-, and 600-grit silicon carbide abrasive paper (SiC sandpaper, Buehler) under water and air abraded with 50- μ m aluminum oxide particles at 1 bar and a distance of 10 mm for 10 seconds. The specimens were stored in dry conditions at room temperature until ready to be bonded.

Bonding and Testing

The cements tested are listed in Table 1. A sample size of 12 specimens per study group (n=12) were

prepared. All cement systems were mixed and applied according to the manufacturer's recommendations for each substrate. For those cements requiring the use of a primer, as for Multilink Automix, the corresponding primer (Monobond Plus, Ivoclar Vivadent, Amherst, NY, USA) was used before application of the cement. All bonding procedures were carried out in a temperature-, humidity-, and light-controlled room with overhead lighting that used orange filters to avoid polymerization of the materials due to ambient light photoactivation. To avoid bias during the bonding procedures, study groups were randomized.

The SBS for each cement-substrate combination was tested at 15 minutes and 24 hours in both SP and LP setting reactions. The specimens were secured using a specially fabricated jig (Bonding jig, Ultradent, South Jordan, UT, USA) with a cylindrical mold of 2.38 mm in diameter. The corresponding cement was injected into the cylindrical mold, which was not filled to the top. For the LP groups, specimens were polymerized following manufacturer's recommendations with a light curing unit (Bluephase C8, Ivoclar Vivadent). A minimum power density of 800 mW/cm² was ensured by periodically monitoring the unit's output with a radiometer (Demetron, Kerr, Orange, CA, USA). All specimens were stored at 37°C and 100% humidity until ready to be tested.

Shear bond strength was measured using a testing machine (Ultratester, Ultradent) at a test speed of 1 mm/min. A notched crosshead designed to match the diameter of the bonded specimen was used to apply the testing load (Figure 1). Specimens were stabilized in a testing jig, which was free to move to facilitate positioning under the load. The

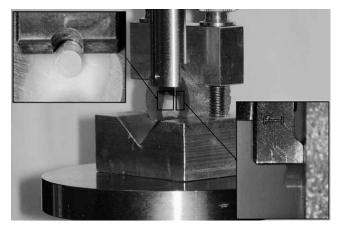


Figure 1. Shear bond strength universal testing machine with a notched cross-head matching the diameter of the bonded specimen.

test base was then positioned so that the notched crosshead was placed against the specimen surface, and the notch was fitted on the diameter of the bonded specimen. The load required to debond the specimen was recorded and expressed in MPa by dividing the load by the surface area of the bonded specimen, and the mean SBS for each study group was calculated.

Statistical Analyses

A Student t-test was used to determine whether significant differences existed between setting reactions (SP vs LP) and between testing times (15 minutes vs 24 hours) regardless of other variables. A one-way analysis of variance (ANOVA) was performed to evaluate whether significant differences existed among cements stratified by setting reaction and testing time irrespective of the substrate. A multifactorial analysis with generalized linear model was used to evaluate the effect of multiple covariates (substrate, cement, and setting reaction) and all their interactions on SBS at each testing time. Post hoc analysis with Tukey test was conducted to explore the presence of significant differences between specific substrate-cement combinations for each testing condition (setting reaction and testing time). For each substrate-cement combination, we also reported pretesting failures or samples spontaneously debonded prior to testing. A significance level of 0.05 was used for all tests. All statistical analysis was performed with Statistical Package for Social Sciences (SPSS) version 16.0 (SPSS Inc. Chicago, IL, USA).

RESULTS

Effect of the Setting Reaction

Student t-test revealed significantly higher mean SBS values for LP relative to SP groups at both testing times irrespective of substrate-cement interactions (p<0.001). At 15 minutes, mean SBS values were 11.4 ± 0.5 and 3.3 ± 0.1 for LP and SP, respectively. At 24 hours, mean SBS values were 15.8 ± 0.8 and 11.6 ± 0.4 MPa for LP and SP, respectively. However, when specific interactions were considered by a one-way ANOVA, a few exceptions were observed. FujiCEM did not show significant differences between SP and LP modes when evaluated at either 15 minutes (p=0.40) or at 24 hours (p=0.54). All self-adhesive resin cements showed significant differences between SP and LP modes irrespective of the substrate at both testing times. The only exception was RelyX Unicem, which

Cements	SP/15 min Mean (SE)	SP/24 h Mean (SE)	LP/15 min Mean (SE)	LP/24 h Mean (SE)
RelyX Unicem	1.5 (0.1) ^a	18.5 (0.8) ^a	11.4 (0.6) ^a	18.7 (1.3) ^{a,}
Maxcem Elite	5.1 (0.2) ^b	11.7 (0.6) ^b	10.6 (0.4) ^a	16.8 (0.5) ^b
Multilink Automix	2.6 (0.3)°	8.9 (0.3)°	19.0 (1.1) ^b	21.8 (2.1) ^a
FujiCEM Automix	4.0 (0.1) ^d	5.3 (0.3) ^d	4.0 (0.2) ^c	5.6 (0.3) ^c

demonstrated no differences between SP and LP modes when evaluated at 24 hours (p=0.89).

Effect of the Testing Time

Student t-test also demonstrated significantly higher mean SBS values at 24 hours relative to 15 minutes for both setting reactions irrespective of substrate-cement combination (p<0.001). However, when specific substrate-cement interactions were considered in a *post hoc* analysis, a few exceptions showed no significant differences between testing times. In LP mode, no significant differences were detected between 15 minutes and 24 hours for Multilink

Automix bonded to base metal (p=0.87) and noble metal (p=0.12), RelyX Unicem bonded to ceramic (p=0.97), and FujiCEM bonded to base metal (p=0.47) and zirconia (p=0.99). In SP mode, no significant differences between 15 minutes and 24 hours were found for FujiCEM bonded to zirconia (p=0.13) and ceramic (p=0.96).

Effect of the Cement

Table 2 summarizes mean SBS values for the different cements under the different testing conditions. One-way ANOVA revealed significant differences in mean SBS values among cements

Table 3: Generalized Linear Model for Multiple Comparisons of Substrates, Cements, and Setting Reactions, as Well as Their Interactions (n=941)

Variable	Type III Sum of Squares	df	Mean Square	F	Sig			
Intercept	100378.499	1	100378.499	3523.329	0.000			
Cement	10126.694	3	3375.565	118.484	0.000			
Substrate	7018.774	4	1754.693	61.590	0.000			
Setting reaction	9268.924	1	9268.924	325.343	0.000			
Cement * substrate	7024.988	12	585.416	20.548	0.000			
Cement * setting reaction	6245.785	3	2081.928	73.077	0.000			
Substrate * setting reaction	3994.508	4	998.627	35.052	0.000			
Cement * substrate * setting reaction	5197.472	12	433.123	15.203	0.000			
Abbreviations: Adjusted R ² ,0.643; df, degree of freedom; F, F statistic; Sig, significance level.								

irrespective of substrate for each setting reaction and testing time (p < 0.001). Post hoc analysis with Tukey test revealed that all cements were significantly different from each other (p<0.001) except for RelyX Unicem and Maxcem Elite in LP mode at 15 minutes (p=0.85). In LP mode at 24 hours, no significant differences were shown between RelyX Unicem and Multilink Automix (p=0.34) and RelyX Unicem and Maxcem Elite (p=0.71). The highest mean SBS was shown for RelyX Unicem and Multilink Automix in LP mode at 24 hours with values of 18.7 and 21.8 MPa, respectively.

Effect of Multiple Factors

Table 3 summarizes the results from the multifactorial analysis. The generalized linear model revealed that all factors (cement, substrate, and setting reaction) as well as all of their interactions were found to have a significant effect in the SBS (p < 0.001).

Mean SBS values for the different cement-substrate combinations under different testing conditions are summarized in Figure 2 and Table 4. Significant differences were evidenced between cements for each substrate under the same testing conditions (letters in Table 4), and between substrates for each cement under the same testing conditions (letters in Figure 1). As shown in Figure 1. resin specimens bonded to RelvX Unicem and Multilink Automix at 24 hours in LP mode showed significantly higher SBS than the other substrates (p<0.05). Resin specimens bonded to RelyX Unicem also demonstrated significantly higher SBS than the other substrates at 24 hours in SP mode (p < 0.05). Base metal bonded to Maxcem Elite showed significantly higher SBS than the other substrates at 24 hours (p < 0.05). Post hoc analysis with Tukey test shown in Table 4 revealed that FujiCEM Automix consistently showed significantly lower mean SBS than all other self-adhesive resin cements (p < 0.05). The only exception was when the different substratecement combinations were evaluated in SP mode at 15 minutes.

Pretesting failures or specimens spontaneously debonded prior to testing were observed in some groups. As shown in Table 4, Rely X Unicem and Multilink Automix showed debonding of only one specimen from noble and base metal, respectively,

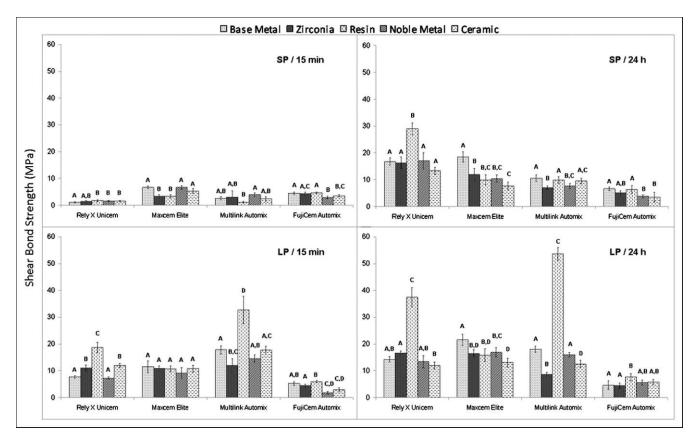


Figure 2. Mean shear bond strengths in MPa for the different study groups. For each time, polymerization method, and cement, same letter indicates substrates that are not statistically different under the same testing conditions (setting reaction and testing time).

Table 4: Mean Shear Bond Strengths in MPa for Each of the Tested Groups With Number of Pretest Failures or Samples Spontaneously Debonded Prior to Testing*

Substrate	Time	Mode	RelyX Unicem		Maxcem Elite		Multilink Automix		FujiCEM Automix	
			Mean (SE)	No. PTF	Mean (SE)	No. PTF	Mean (SE)	No. PTF	Mean (SE)	No. PTF
Base metal	15 min	SP	1.0 (0.1) ^a	0	6.7 (0.2) ^b	0	2.6 (0.3)°	0	4.5 (0.2) ^d	0
	_	LP	7.6 (0.2) ^a	0	11.5 (1.1) ^b	0	17.8 (0.8)°	0	5.2 (0.3) ^d	0
	24 h	SP	16.8 (0.7) ^a	0	18.5 (1.0) ^a	0	10.6 (0.6) ^b	0	6.6 (0.4) ^c	0
	_	LP	14.3 (0.5) ^a	0	21.6 (1.1) ^b	0	18.0 (0.5) ^c	1	4.6 (0.8) ^d	0
Noble metal	15 min	SP	1.6 (0.1) ^a	0	6.6 (0.4) ^b	0	4.0 (0.3) ^c	0	2.9 (0.2) ^d	0
	_	LP	7.2 (0.2) ^a	1	9.2 (1.0) ^a	0	14.6 (0.7) ^b	0	1.8 (0.2) ^c	2
_	24 h	SP	17.1 (1.6) ^a	0	10.4 (0.7) ^b	0	7.7 (0.5) ^b	0	3.8 (0.4) ^c	0
	_	LP	13.4 (1.2) ^a	0	16.9 (0.8) ^b	0	15.9 (0.4) ^b	0	5.6 (0.6) ^c	2
Zirconia	15 min	SP	1.4 (0.1) ^a	0	3.3 (0.3) ^{a,b}	0	3.0 (1.3) ^{a,b}	0	4.3 (0.3) ^b	1
	_	LP	11.0 (0.6) ^a	0	10.9 (0.5) ^a	0	12.1 (1.3) ^a	0	4.4 (0.3) ^b	0
_	24 h	SP	16.2 (1.2) ^a	0	11.9 (1.1) ^b	0	7.1 (0.3)°	0	5.1 (0.4) ^c	0
	_	LP	16.6 (0.4) ^a	0	16.5 (0.7) ^a	0	8.7 (0.4) ^b	0	4.4 (0.5)°	0
Ceramic	15 min	SP	1.7 (0.1) ^a	0	5.3 (0.4) ^b	0	2.4 (0.5) ^a	0	3.6 (0.1) ^c	0
_	_	LP	12.0 (0.4) ^a	0	10.8 (0.7) ^a	0	17.6 (0.8) ^b	0	2.9 (0.1) ^c	2
	24 h	SP	13.4 (0.7) ^a	0	7.7 (0.7) ^b	0	9.5 (0.5) ^b	0	3.6 (0.9) ^c	7
	_	LP	12.0 (0.7) ^a	0	13.0 (0.9) ^a	0	12.5 (0.7) ^a	0	5.8 (0.4) ^b	0
Resin	15 min	SP	1.7 (0.2) ^a	0	3.4 (0.3) ^b	2	1.1 (0.2) ^a	0	4.6 (0.1) ^c	0
	_	LP	18.6 (1.0) ^a	0	10.7 (0.6) ^b	0	32.7 (2.6) ^c	0	6.0 (0.2) ^d	0
_	24 h	SP	29.0 (1.2) ^a	0	10.0 (1.1) ^b	0	9.9 (0.7) ^b	0	6.5 (0.7) ^c	0
	_	LP	37.4 (1.8) ^a	0	15.8 (1.3) ^b	0	53.6 (1.2) ^c	0	7.6 (0.7) ^d	0

Abbreviations: LP, light-polymerizing mode; PTF, Pretesting failures; SE, standard error; SP, self-polymerizing.

* Groups with the same superscript letter indicate cements that are not significantly different for each substrate under the same conditions (Tukey test).

and Maxcem Elite showed debonding of two resin specimens. FujiCEM showed a much higher rate of pretesting failures predominantly to noble metal and ceramic, with four and nine samples debonded, respectively.

DISCUSSION

This study evaluated the shear bond strength of a number of cements bonded to a variety of prosthodontic substrates tested at different times (15 minutes and 24 hours) and undergoing different setting reactions (SP and LP). The null hypothesis was rejected as significant differences in SBS were detected among cements, between SP and LP modes, and between 15 minutes and 24 hours. Furthermore, results from the multiple comparisons revealed that all interactions between the tested factors were also found to be significant with certain combinations of cement, substrate, and setting reaction showing improved bond strengths.

Effect of the Setting Reaction

Overall, significantly higher SBS were demonstrated when specimens were light-activated compared to values generated when the cements were allowed to self-polymerize. Similar findings have been reported in the literature. 4,16-18 Light polymerization yielded improved SBS of the three selfadhesive resin cements, which was also shown to be dependent on the cement. Different monomer composition and polymerization conditions have been shown to alter the degree of conversion, resulting in variations in bond strength results. 19 FujiCEM was the only cement that did not show differences between setting reactions at either 15 minutes or 24 hours. A recent report demonstrated that RMGIC acid-base and visible light polymerization reactions inhibit one another during the early phases of setting,20 which may help explain why no differences were observed between SP and LP mode for FujiCEM. With the exception of FujiCEM, all substrate-cement combinations demonstrated higher SBS when light-activated. The only exception was RelyX Unicem, which demonstrated no differences between SP and LP modes when evaluated at 24 hours. Both RMGIC and selfadhesive resin cements set by an acid-base reaction as well as a free radical polymerization reaction. While dual polymerizing systems are known to compensate for light attenuation through the thickness of the indirect restoration,²¹ it has been shown that, compared to RMGIC, resin cements typically exhibit higher bond strengths when lightactivated.4 A number of studies have reported higher degree of conversion under light-polymerization conditions for resin-based materials. 17,18,22

Effect of the Testing Time

The SBS of all cements were also shown to be higher after 24 hours relative to 15 minutes irrespective of the substrate and type of setting reaction. This might have been the result of the continued postirradiation polymerization reaction known to take place after the reaction is initiated and that lasts for up to 24 hours. 23,24 With the exception of a few groups, most substrate-cement combinations demonstrated an increase in SBS after 24 hours when evaluated in both setting reactions. In general, these differences remained significant and were more apparent for the three self-adhesive resin cements than the RMGIC. Similar findings of increased bond strength after 24 hours have been reported previously for different cements.²⁵ Fuji-CEM showed only a slight increase in mean SBS from 15 minutes to 24 hours. Some FujiCEM groups showed no change (FujiCEM bonded to ceramic in SP mode and FujiCEM bonded to zirconia in LP mode) or even a decrease in SBS values (FujiCEM bonded to base metal in LP mode) after 24 hours. Although our study did not formally measure the extent of the polymerization reaction, the slight-tono increase in SBS values for FujiCEM after 24 hours suggests an apparent contradiction with previous studies, which have demonstrated that the RMGIC acid-base reaction continues overtime if undisturbed. 26,27

Effect of the Cement and Multiple Interactions

Significantly higher SBS were evidenced for the three self-adhesive resin cements compared to FujiCEM for all testing conditions except in SP mode at 15 minutes. This is in agreement with previous studies, which have shown higher bond strengths for resin-based cements relative to RMGIC.^{4,28} The similar SBS values for all cements when evaluated in SP mode at 15 minutes may have been the result of a slow initial cross-linking of the resin-based materials when they were allowed to self-polymerize. Multilink Automix and RelyX Unicem vielded the highest SBS irrespective of the substrate. A recent study demonstrated similar findings, with RelyX Unicem showing higher bond strengths compared to FujiCEM when bonded to base and noble metals, ceramic, and zirconia substrates.⁵ Another study by Zhang and Degrange²⁹ showed higher bond strengths for Multilink Automix

compared to other self-adhesive resin cements regardless of the restorative substrate. The same study also found that the bond strengths for many of the tested cements were dependent on the nature of the restorative substrate.²⁹ This is coincident with the results from our study which demonstrated that the interactions between cement, substrate, and setting reaction were also found to have a significant effect in the bond strength. The synergistic behavior whereby certain combinations of cement, substrate and setting reaction are more favorable than others indicates that the selection of the luting cement should be partially dictated by the substrate and the setting reaction. Resin bonded to Multilink Automix and RelyX Unicem in LP mode, and resin bonded to RelyX Unicem in SP mode showed higher SBS values than any of the other combinations in all testing conditions. Similar findings have been reported for RelyX Unicem. 4 Compatibility between the resinous components in the matrix of cements Multilink Automix and RelyX Unicem and those of composite Z100 may have been partially responsible for the observed results. Similarly, Maxcem Elite showed higher SBS values to base metal relative to all other substrates in all testing conditions. This could have been the product of the surface oxides known to make the base metal more reactive by providing potential for chemical bonding.⁵ Presumably, a greater chemical affinity between the components of self-adhesive resin cements and those of specific prosthodontic substrates may have been responsible for the observed results. However, only general estimations can be made based on the information provided by the manufacturer because specific details regarding the material's chemical composition are proprietary. As recommended in the cements' directions for use, air abrasion with 50-µm aluminum oxide particles was used for surface roughening of all the substrate materials prior to bonding. Since the manufacturers do not specify additional surface treatment before application of the cement, no further surface treatments such as acid-etching or silanization were used as this might have led to different results. Only when bonding with Multilink Automix, was Monobond Plus primer used after air abrasion as per manufacturer's instructions.

As self-adhesive cements continue to gain acceptance in the market, large comparative studies are needed to evaluate their behavior when bonded to a variety of prosthodontic substrates and tested under different testing conditions. Bond strength studies represent valuable initial screening tests to assess

the overall behavior and predict future clinical performance of the materials and techniques under investigation. However, care should be exercised when extrapolating the results obtained from laboratory studies to the expected clinical outcomes as in vitro tests are subject to a number of limitations. In the present study, a 24-hour immersion in a 37°C water bath was used prior to bond strength testing since this represents the standard short-term storage protocol recommended by the International Organization for Standardization (ISO/TR 11405).³⁰ Although the effects of thermal cycling and longterm storage on the bond strength were not evaluated as a part of this investigation, they are important in the simulation of clinical conditions and should be investigated in future laboratory studies incorporating multiple variables such as those included in the present study. Furthermore, a direct comparison among studies seems unfair since a number of aspects relative to the design and methodology are known to vary between studies. Since the aim of our study was to isolate specific interactions between the tested cements and different substrates, a simplified interfacial design was used, whereby the luting cement was directly bonded onto the substrate. A different methodology used in some studies involves two substrates (adherends) which are joined together by a luting cement (adhesive).31 While this design resembles more closely the clinical situation, whereby a prepared tooth receives a laboratory-processed restoration, it represents a more complex interface since three different materials are joined together making it difficult to isolate the specific interactions taking place between the different components of the interface and perhaps compromising the validity of the results.

Further research is needed to validate the longterm behavior of the different substrate-cement combinations when tested in a variety of testing conditions. No conclusions can be drawn based solely on the results from bond strength studies. Combining the results from bond strength studies with those from microleakage and marginal adaptation studies may provide a more comprehensive assessment of the performance of the systems under investigation. Furthermore, inclusion of failure mode analysis routinely in bond strength studies may significantly contribute to a more accurate interpretation of the obtained results, as well as facilitate a better understanding of the mechanical behavior and stress distribution of adhesive interfaces during failure.

CONCLUSIONS

Within the limitations of the present *in vitro* study, the following conclusions may be drawn:

- 1. The performance of the cements was greatly dependent on the type of setting reaction, with light-polymerized mode displaying significantly higher bond strengths than self-polymerized mode. The performance of the cements was also dependent on testing time. After 24 hours, all cements matured showing higher bond strengths than initial values obtained at 15 minutes.
- 2. Overall, self-adhesive resin cements demonstrated higher bond strengths than RMGIC FujiCEM Automix irrespective of the substrate for all testing conditions. The best performance was achieved for RelyX Unicem at 24 hours (SP and LP modes) and Multilink Automix in LP mode (15 minutes and 24 hours).
- 3. The bond strength of the cements also varied depending on the prosthodontic substrate, indicating that selection of the cement should be dictated partially by the substrate. Overall, Multilink Automix and RelyX Unicem demonstrated higher SBS when bonded to resin, and Maxcem Elite demonstrated higher SBS when bonded to base metal.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 1 May 2012)

REFERENCES

- 1. Abo-Hamar SE, Hiller KA, Jung H, Federlin M, Friedl KH. & Schmalz G (2005) Bond strength of a new universal self-adhesive resin luting cement to dentin and enamel Clinical Oral Investigations 9(3) 161-167.
- 2. Hikita K, Van Meerbeek B, De Munck J, Ikeda T, Van Landuyt K, Maida T, Lambrechts P, & Peumans M (2007) Bonding effectiveness of adhesive luting agents to enamel and dentin Dental Materials 23(1) 71-80.
- 3. Sindel J, Frankenberger R, Kramer N & Petschelt A (1999) Crack formation of all-ceramic crowns dependent on different core build-up and luting materials Journal of Dentistry 27(3) 175-181.
- 4. Piwowarczyk A, Lauer HC, & Sorensen JA (2004) In vitro shear bond strength of cementing agents to fixed prosthodontic restorative materials Journal of Prosthetic Dentistry **92(3)** 265-273.
- 5. Capa N, Özkurt Z, Canpolat C, & Kazazoglu E (2009) Shear bond strength of luting agents to fixed prosthodon-

- tic restorative core materials Australian Dental Journal **54(4)** 334-340.
- 6. Piwowarczyk A, Bender R, Ottl P, & Lauer HC (2007) Long-term bond between dual-polymerizing cementing agents and human hard dental tissue Dental Materials **23(2)** 211-217.
- 7. Piwowarczyk A, Lauer HC, & Sorensen JA (2003) Dentin shear bond strength of various luting cements Journal of Dental Research 82(Special Issue C) p 501.
- 8. Piwowarczyk A, Lindemann K, Zipprich H, & Lauer HC (2003) Long-term shear bond strength of luting cements to dentin Journal of Dental Research 82(Special Issue **B**) Abstract #1456 p B-194.
- 9. Holderegger C, Sailer I, Schuhmacher C, Schläpfer R, Hämmerle C, & Fischer J (2008) Shear bond strength of resin cements to human dentin Dental Materials 24(7) 944-950.
- 10. Furuchi M, Oshima A, Ishikawa Y, Koizumi H, Tanoue N, & Matsumura H (2007) Effect of metal priming agents on bond strength of resin-modified glass ionomers joined to gold alloy Dental Materials Journal 26(5) 728-732.
- 11. Matsumura H, Yanagida H, Tanoue N, Atsuta M, & Shimoe S (2001) Shear bond strength of resin composite veneering material to gold alloy with varying metal surface preparations Journal of Prosthetic Dentistry **86(3)** 315-319.
- 12. Yoshida K, Kamada K, Sawase T, & Atsuta M (2001) Effect of three adhesive primers for a noble metal on the shear bond strengths of three resin cements Journal of Oral Rehabilitation 28(1) 14-19.
- 13. Cobb DS, Vargas MA, Fridrich TA, & Bouschlicher MR (2000) Metal surface treatment: Characterization and effect on composite-to-metal bond strength Operative Dentistry **25(5)** 427-433.
- 14. Guarda GB, Gonçalves LS, Correr AB, Moraes RR, Sinhoreti MAC, & Correr-Sobrinho L (2010) Luting glass ceramic restorations using a self-adhesive resin cement under different dentin conditions Journal of Applied Oral Science 18(3) 244-248.
- 15. Aguiar TR, Di Francescantonio M, Ambrosano GMB, & Giannini M (2010) Effect of curing mode on bond strength of self-adhesive resin luting cements to dentin *Journal of* Biomedical Materials Research. Part B, Applied Biomaterials 93(1) 122-127.
- 16. De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, & Van Meerbeek B (2004) Bonding of an auto-adhesive luting material to enamel and dentin Dental Materials **20(10)** 963-971.
- 17. Vrochari AD, Eliades G, Hellwig E, & Wrbas KT (2009) Curing efficiency of four self-etching, self-adhesive resin cements Dental Materials 25(9) 1104-1108.
- 18. Cadenaro M, Navarra CO, Antoniolli F, Mazzoni A, Di Lenarda R, Rueggeberg FA, & Breschi L (2010) The effect of curing mode on extent of polymerization and microhardness of dual-cured, self-adhesive resin cements American Journal of Dentistry 23(1) 14-18.
- 19. Ferracane JL, & Greener EH (1986) The effect of resin formulation on the degree of conversion and mechanical properties of dental restorative resins Journal of Biomedical Materials Research 20(1) 121-131.

20. Berzins DW, Abey S, Costache MC, Wilkie CA, & Roberts HW (2010) Resin-modified glass-ionomer setting reaction competition *Journal of Dental Research* **89(1)** 82-86.

- Rueggeberg FA, & Caughman WF (1993) The influence of light exposure on polymerization of dual-cure resin cements Operative Dentistry 18(2) 48-55.
- 22. Mendes LC, Matos IC, Miranda MS, & Benzi MR (2010) Dual-curing, self-adhesive resin cement: Influence of the polymerization modes on the degree of conversion and microhardness *Materials Research* 13(2) 171-176.
- 23. Peters AD, & Meiers JC (1996) Effect of polymerization mode of a dual-cured resin cement on time-dependent shear bond strength to porcelain *American Journal of Dentistry* **9(6)** 264-268.
- 24. Eliades GC, Vougiouklakis GJ, & Caputo AA (1987) Degree of double bond conversion in light-cured composites *Dental Materials* **3(1)** 19-25.
- 25. Faria-e-Silva AL, Fabiao MM, Arias VG, & Martins LR (2010) Activation mode effects on the shear bond strength of dual-cured resin cements *Operative Dentistry* **35(5)** 515-521.
- Wan ACA, Yap AUJ, & Hastings GW (1999) Acid-base complex reactions in resin-modified and conventional

- glass ionomer cements Journal of Biomedical Materials Research 48(5) 700-704.
- Young AM, Rafeeka SA, & Howlett JA (2004) FTIR investigation of monomer polymerisation and polyacid neutralisation kinetics and mechanisms in various aesthetic dental restorative materials *Biomaterials* 25(5) 823-833.
- Ernst CP, Aksoy E, Stender E, & Willershausen B (2009)
 Influence of different luting concepts on long term retentive strength of zirconia crowns American Journal of Dentistry 22(2) 122-128.
- 29. Zhang CX, & Degrange M (2010) Shear bond strengths of self-adhesive luting resins fixing dentine to different restorative materials *Journal of Biomaterials Science*. *Polymer Edition* **21(5)** 593-608.
- International Organization for Standardization (2003) ISO/TS 11405 Dental Materials—Testing of Adhesion to Tooth Structure International Organization for Standardization, Geneva.
- 31. Ernst CP, Doz P, Cohnen U, Stender E, & Willershausen B (2005) *In vitro* retentive strength of zirconium oxide ceramic crowns using different luting agents *Journal of Prosthetic Dentistry* **93(6)** 551-558.